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Synthesis, characterization and comparison of sediment electro-codeposited nickel–micro and nano SiC composites

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ABSTRACT

Nickel–silicon carbide composites were produced using 1 μm and 50 nm size powders from a conventional Watt's bath using tetra methyl ammonium hydroxide as the surfactant. Sediment codeposition technique with horizontal electrodes was used. The effect of silicon carbide concentration and bath operating parameters on the volume percents and deposition rates of coatings obtained with the two different particles was studied. Substantial improvements in mechanical properties such as hardness, wear resistance, scratch resistance and roughness were obtained with the nanocomposite material, as compared with composites containing microsized particles.

1. Introduction

The codeposition of ceramic particles with metals from electrolytic solutions for the production of metal matrix composite coatings is a broad area of research interest as these coatings are used for a wide variety of industrial applications, especially in the area of tribology [1–5].

This process has the advantages of low working temperatures, low cost, easy maintenance, ability to produce composite coatings and versatility with different combination of properties by just changing the electroplating conditions [6]. The low processing temperature (around room temperature) minimizes interdiffusion or chemical reaction between the substrate and coating species. The film thickness can be accurately controlled by monitoring the consumed charge and the composition can be tailored by the electrical applied profile and bath composition [7]. Moreover, it is quickly scaled up to industrial production, offering an inexpensive method to produce large area samples.

The embedded second-phase hard particles impart special physical and mechanical properties to these coatings [8]. Uniform dispersion of the second-phase hard particles leads to the improvement of the mechanical and tribological, properties of the coatings [9,10].

The process can be carried out using either Conventional Electro-Co-Deposition technique (CECD) in which the electrodes are positioned vertically in the plating cell or by Sediment Electro-Co-Deposition (SECD) in which the electrodes are positioned horizontally one over the other with sufficient inter-electrode distance so that the particle settle on the electrode surface as sediment on the cathode as the metal deposition progresses [11,12]. The latter has the advantage of yielding considerably higher vol.% incorporation of particles in the deposit compared to the CECD technique for a given vol.% of particles in the solution. This has the advantage of conserving the costly insoluble powders especially those with a very fine size. Schematic of the CECD and SECD techniques are shown in Fig. 1.

With the emergence of nanostructured materials, research on the production of nanocomposite coatings by electrolytic co-deposition has received much interest due mainly to the fact that these coatings can enhance properties like hardness [13,14], wear resistance [15], strength [16,17], corrosion resistance, oxidation resistance and self-lubrication, etc., to a plated surface [18,3,19]. Materials are considered nanosized when one of the components dimensions are in the nanometer scale, with typical dimensions smaller than 100 nm. A variety of nanosized particles ranging from 4 nm to 800 nm diameters, have been successfully incorporated

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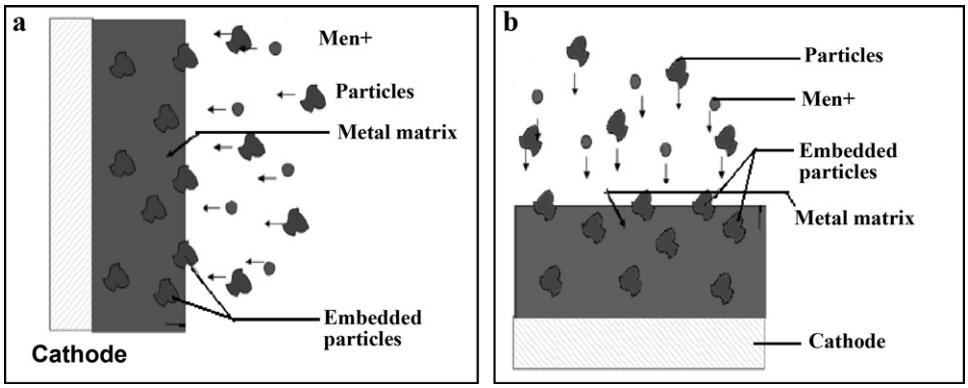

Fig. 1. Schematic of the codeposition techniques. (a) CECD and (b) SECD.

into metallic electrodeposits [3,20–22]. The metals mostly used are copper and nickel. The most studied system has been the Ni–SiC due to its potential technological applications [8,14,22–27].

The inclusion of nanosized particles, especially those below 100 nm, into metal deposits is somewhat difficult compared to the micron size particles [28,29] and dependent on many process parameters, like the electrolysis conditions (composition and agitation of the electrolytic bath, presence of additives, temperature, pH), the electrical profile and the particle properties (type, size, shape, surface charge, concentration and dispersion in the bath) [4,14,18,30–34]. In addition, the agglomeration of these particles in the electrolyte would result in their agglomeration in the codeposited layer [13]. In other words, non-homogeneously distributed particles would appear in the matrix, which would be detrimental to the mechanical property of the deposit [35]. Furthermore the co-deposition of a sufficient amount of nonagglomerated particles should lead to production of harder and more corrosion resistant coatings [21]. Various additives have been studied to reduce agglomeration of particles [29]. Further, the codeposition of β-SiC is more difficult than α-SiC [18].

Studies have also been reported on the influence of operating parameters on the codeposition of nano-SiC in the nickel matrix [22,23,36]. Gyftou et al. [8] have reported the co-deposition mechanism of micro- and nano-SiC particles incorporated in nickel matrix. The mechanical properties of Ni–SiC nanocomposites from modified Watt's bath have been studied by Zimmermann et al. [23]. Garcia et al. [14] have made Ni–SiC composite coatings using SiC particles with three different sizes (5, 0.7 and 0.3 μm) and investigated their sliding wear resistance. The electro-codeposition process and hence the structure, the morphology and the properties of the composite coatings is affected by the electrodeposition parameters. Liu et al. [37] have analyzed the tensile strength and microstructure of Ni–SiC(micro) composites obtained from sulphamate electrolyte. Super-plasticity behavior of Ni–SiC (nano) composite has been studied by Chen et al. [38]. Wear corrosion properties of nano-structured SiC–nickel composite coatings obtained from Watt's bath have also been investigated [31,34].

The properties of Ni–SiC composite coatings have been improved by producing it as gradient coatings [38–40]. The advantages gained by using pulse technique and triangular waveform have been discussed [8,36,40–44]. Nanocomposites have been produced by electroless technique also [45–49].

Ni–SiC composite is already a commercial reality and is widely used in automobile and textile components. With the increasing availability of nanoparticles, the interest on composite electroplating is continuously growing, with the major challenge being the achievement of high co-deposition rates, homogenous distribution of the particles in the metallic matrix and ability to produce them with consistent and controlled composition and properties. Industrial exploitation of nanocomposites is possible only by optimizing various parameters including deposition conditions. The available data are varied due to difference in the nature of bath, type of SiC (α or β) and its particle size, additives, current mode, or testing/analyzing methods adopted, etc. Also, none of the above data is available on Ni–SiC nanocomposites produced by SECD technique in which the conditions are different from those used in CECD technique.

The aim of this work is to optimize operating conditions to produce Ni–SiC (~50 nm) nanocomposites with maximum hardness, wear resistance, scratch resistance and roughness by SECD technique and compare the results with those of Ni–SiC microcomposite (~1 μm) prepared using the same technique. This is to justify the advantages of using nanocomposites over the relatively cheap and less cumbersome micro composites.

This paper deals with the laboratory scale preparation and characterization of nickel–β SiC the above composites of micro and nano sizes using SECD technique. The effects of plating parameters such as SiC content in the plating bath, pH of the electrolyte, current density and stirring speed on the vol.% incorporation of the particles are studied and their properties like hardness, wear resistance, scratch resistance, roughness and microstructure are evaluated. The results will be used for mathematical modeling experiments [50] which will be dealt with in our next paper.

2. Experimental

2.1. Electrolyte preparation

The plating solution used was a standard Watts' nickel solution. The composition of the plating solution and the plating parameters are given in Table 1. The bath was prepared using laboratory grade reagents, and purified in the conventional manner [51]. Tetramethyl ammonium hydroxide (TMAH) was prepared as an aqueous stock solution and used as the surfactant. Electrolyte pH was adjusted to 4 electrometrically using dilute sodium hydroxide or sulphuric acid.

Table 1
Bath composition and conditions used for deposition.

Constituent	Concentration, g/l
Nickel sulphate 6 H_2O	250
Nickel chloride 6 H_2O	30
Boric acid	40
pH	2–5
Current density	1–3 A/dm^2
Temperature	30–60 °C
Agitation speed	Magnetic stirring, 200–600 rpm